

Integran Technologies Inc

I38003 Al/Sg

#6208 German Claims (Allowed)

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1. Process for cathodically electrodepositing a selected metallic material on a permanent or temporary substrate in nanocrystalline form with an average grain size of less than 100 nm using pulse electrodeposition at a deposition rate of at least 0.05 mm/h, comprising:

- 10 - providing an aqueous electrolyte containing ions of said metallic material,
- maintaining said electrolyte at a temperature in the range between 0 to 85°C,
- agitating the electrolyte at an agitation rate in the range of 1 to 750 ml per min and per A,
- 15 - providing an anode and a cathode in contact with said electrolyte,
- passing single or multiple D.C. cathodic-current pulses between said anode and said cathode at pulsed intervals during which said current passes for a t_{on} -time period in the range of about 0,1 to 50 msec and does not pass for a t_{off} -time period in the range of about 0 to 500 msec,
- 20 - passing single or multiple D.C. anodic-current pulses between said cathode and said anode at intervals during which said current passes for a t_{anodic} -time period in the range of 0 to 50 msec,
- 25 - a duty cycle being in a range of 5 to 100%; and
- whereby the cathodic charge ($Q_{cathodic}$) per interval is always larger than a anodic charge (Q_{anodic}) per interval.

2. Process as claimed in claim 1, characterized in that the single or multiple D.C. cathodic-current pulses between said anode and said cathode have a peak current density in the range of about 0.01 to 20 A/cm².
- 5 3. Process as claimed in claim 2, characterized in that the peak current density of the cathodic-current pulses is in the range of about 0.1 to 20 A/cm², preferably in the range of about 1 to 10 A/cm².
4. Process as claimed in any of claims 1 to 3, characterized in that said
10 selected metallic material is (a) a pure metal selected from the group consisting of Ag, Au, Cu, Co, Cr, Ni, Fe, Pb, Pd, Pt, Rh, Ru, Sn, V, W, Zn, or (b) an alloy containing at least one of the elements of group (a) and alloying elements selected from the group consisting of C, P, S and Si.
- 15 5. Process as claimed in any of claims 1 to 4, characterized in that the t_{on} -time period is in the range of about 1 to about 50 msec, the t_{off} -time period is in the range of about 1 to 100 msec and the t_{anodic} -time period is in the range of about 1 to 10 msec.
- 20 6. Process as claimed in any of claims 1 to 5, characterized in that the duty cycle preferably is in the range of 10 to 95 %, and more preferably is in the range of 20 to 80 %.
7. Process as claimed in any of claims 1 to 7, characterized in that the
25 deposition rate is preferably at least 0.075 mm/h and more preferably at least 0.1 mm/h.
8. Process as claimed in any of claims 1 to 7, characterized by agitating the electrolyte at an agitation rate in the range of 1 to 500 ml/min/A.

9. Process as claimed in claims 1 to 8, characterized by agitating the electrolyte by means of pumps, stirrers or ultrasonic agitation.

10. Process as claimed in any of claims 1 to 9, characterized by a relative motion between anode and cathode.
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11. Process as claimed in claim 10, characterized in that the speed of the relative motion between anode and cathode ranges from 0 to 600 m/min, preferably from 0.003 to 10 m/min.
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12. Process as claimed in claim 10, characterized in that the relative motion is achieved by rotation of anode and cathode relative to each other.
13. Process as claimed in claim 12, characterized by a rotational speed of rotation of anode and cathode relative to each other ranging from 0.003 to 0.15 rpm and preferably from 0.003 to 0.05 rpm.
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14. Process as claimed in claim 10 or claim 11, characterized in that the relative motion is achieved by a mechanized motion generating a stroke of the anode and the cathode relative to each other.
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15. Process as claimed in claim 10 or 14, characterized in that the anode is wrapped in an absorbent separator.
- 25 16. Process as claimed in any of claims 1 to 15, characterized in that said electrolyte contains a stress relieving agent or a grain refining agent selected from the group of saccharin, coumarin, sodium lauryl sulfate and thiourea.

17. Process as claimed in any of claims 1 to 16, characterized in that said electrolyte contains particulate additives in suspension selected from pure metal powders, metal alloy powders or metal oxide powders of Al, Co, Cu, In, Ng, Ni, Si, Sn, V and Zn, nitrides of Al, B and Si, carbon C (graphite or diamond), carbides of B, Bi, Si, W, or organic materials such as PTFE and polymers spheres, whereby the electrodeposited metallic material contains at least 5 % of said particulate additives.
18. Process as claimed in claim 17, characterized in that the electrodeposited metallic material contains at least 10 % of said particulate additives.
19. Process as claimed in claim 17, characterized in that the electrodeposited metallic material contains at least 20 % of said particulate additives.
20. Process as claimed in claim 17, characterized in that the electrodeposited metallic material contains at least 30 % of said particulate additives.
21. Process as claimed in claim 17, characterized in that said electro deposited metallic material contains at least 40 % of said particulate additives.
22. Process as claimed in any of claims 17 to 21, characterized in that the particulate additives average particle size is below 10 μm , preferably below 1,000 nm, more preferably below 500 nm and most preferably below 100 nm.
23. Micro component produced by a pulse electrodeposition process, especially produced by a pulse electrodeposition process as claimed in any of claims 1 to 22, having a maximum dimension of 1 mm, an average grain size equal to or smaller than 1,000 nm, the ratio between the maximum dimension and the average grain size being greater than 10.

24. Micro component as claimed in claim 23, characterized in that the ratio between the maximum dimension of the micro component and the average grain size is greater than 100.

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25. Micro component as claimed in claim 23 or 24, characterized by having a equiaxed micro structure.